# FABRICATION OF CHITOSAN MEMBRANE: THE EFFECT OF EVAPORATION TIMES ON MEMBRANE PERFORMANCE IN OILY WASTE WATER TREATMENT

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A thesis submitted in fulfillment of the requirements for the award of the degree of Bachelor of Chemical Engineering

Faculty of Chemical & Natural Resources Engineering
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"Saya akui bahawa saya telah membaca karya ini dan pada pandangan saya karya ini adalah memadai dari segi skop dan kualiti untuk tujuan penganugerahan Ijazah Sarjana Muda Kejuruteraan Kimia"

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| Tandatangan | : .................................................. |
| Nama Penyelia | : Cik Siti Kholijah binti Abdul Mudalip |
| Tarikh | : 24 March 2008 |

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I declare that this thesis entitled "*Fabrication of chitosan membrane: The effect of evaporation times on membrane performance in oily waste water treatment*" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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*Special dedication to  my beloved mother and father, Valliama D/O Lechumanan and Pachiapan S/O Selayen Koundru, my brother and sister  and all my family members that always inspire, love and stand besides me, my supervisors, my beloved friends, my fellow colleagues, and all faculty members*

*For all your love, care, support, and believe in me.Thank you so much.*

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## ABSTRACT

Fabrication of chitosan membrane and the effect of evaporation times on membrane performance in oily wastewater treatment are studied in this paper. Chitosan homogenous membranes were fabricated by casting a chitosan/acetic acid solution, and then evaporate it for different periods of time, followed by chemically cross-linking with sulfuric acid. The separation tests using the resulting membranes demonstrate that the chitosan membranes are capable of separating water–oil mixtures in wastewater. The optimized conditions for chemical cross-linking of membranes were found to be 0.5M of the cross-linking reagent (sulfuric acid) and 10 min reaction time at ambient temperature. The preparation time of chitosan membranes is the key factor to evaluate performance of separating water–oil. It was observed in orthogonal tests that the effect of membrane preparation time on the separation factor was significant. The highest separation factor was 1170 and it occurred at 120 minutes of evaporation time. The highest separation index also occurred at this period of time. The highest separation index was 1092609. These results suggested, 120 minutes is the most perfect evaporation time to produce an efficient chitosan membrane which can extract oil from waste water. As a conclusion, the present work clearly correlates the separation performance of water-oil by using the chitosan membrane resulting from the different of evaporation time.

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# ABSTRAK

Cara penghasilan membran chitosan dan kesan perbezaan masa pengewapan semasa menyediakan membran untuk memisahkan minyak daripada air sisa telah dikaji di dalam thesis ini. Membran daripada chitosan ini telah dihasilkan malalui pengadukan chitosan/asid asetik dan campuran itu di biarkan mengewap dengan tempoh masa yang berbeza, di ikuti proses pengenyalan membran dengan menggunakan asid sulfurik. Ujian pemisahan menggunakan membran yang dihasilkan menunjukkan membran chitosan mampu mengasingkan campuran air-minyak di dalam air sisa. Kepekatan asid sulfurik yang paling sesuai untuk membran melalui proses pengenyalan ialah 0.5M dan ia dilakukan dalam masa 10 minit pada suhu keadaan bilik. Masa pengewapan membran chitosan merupakan faktor utama untuk mengkaji kecekapan pengasingan air-minyak. Pemerhatian daripada eksperimen-ekssperimen yang telah dijalankan, masa untuk menyediakan membran mempengaruhi nilai faktor pemisahan. Nilai factor pemisah yang paling tinggi ialah 1170 dan ia berlaku selepas 120 minit masa pengewapan. Nilai indeks pemisah yang paling tinggi juga di dapati pada tempoh masa yang sama. Nilai indeks tersebut ialah 1092609. Nilai-nilai ini membuktikan, 120 minit masa pengewapan adalah tempoh masa pengewapan yang paling sesuai untuk menghasilkan membran chitosan yang mampu memisahkan minyak daripada air sisa. Kesimpulanya daripada ujikaji ini ialah, masa pengewapan yang berbeza merupakan satu factor utama untuk memisahkan minyak daripada air.

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## LIST OF ABBREVIATIONS

BOD - Biochemical oxygen demand

DAF - Dissolved air flotation

COD - Chemical oxygen demand

µm - Micro meter

nm - Nano Meter

NaCl - Sodium chloride

RO - Reverse osmosis

GFD - gallons per square foot per day

HCL -Acid hydrochloric

$H_2SO_4$ - Acid Sulfuric

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# CHAPTER 1

# INTRODUCTION

## 1.1 Research Background

Wastewater is sewage and water that has been used for various purposes around the community. Unless properly treated, wastewater can harm public health and the environment. Fatty organic materials from animals, vegetables, and petroleum are not quickly broken down by bacteria and can cause pollution in receiving environments. When large amounts of oils are discharged to receiving waters from community systems, they increase biochemical oxygen demand (BOD) and they may float to the surface and harden, causing aesthetically unpleasing conditions. They also can trap trash, plants, and other materials, causing foul odors, attracting flies and mosquitoes and other disease vectors. In some cases, too much oil causes septic conditions in ponds and lakes by preventing oxygen from the atmosphere from reaching the water (Mohr, 1989).

Oil and water separation covers a broad spectrum of industrial process operations. There are many techniques employed depending on the situation. This summary will address those separations, which are suited to the chitosan membrane technology. The oily wastewater application can be broken down into categories determined by the type of user and the oil-water separation desired. There is a saying: “Oil and Water don’t mix”. This is true, but they can exist as an emulsion. Oil is not soluble in water but it can exist evenly dispersed as globules in water. The concentration of these globules is a function of mixing or stirring. If allowed to stand the emulsion will

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separate because oil is lighter than water, although, some amount of oil globules will remain in the water. Another interesting fact is that this emulsion can exist two ways. If the concentration of Oil is less than 50%, the water will be the suspension fluid and the oil will be the globule. A phase transition occurs if the oil content is more than 50%. When this happens, the oil is the suspension fluid and the water forms globules. For this reason, hydrophilic membrane separations will be possible only when the oil content is less than 50 % (Zakaria, 1994).

There are several ways for separation of oil from waste water such as centrifuge, rotary drum vacuum filter, dissolved air flotation (DAF), slope plate clarifiers, biological treatment, evaporators and gravity separating devices. Below are some descriptions of the methods:

### 1.1.1 Centrifuge

Uses large horsepower motors and because of the number of moving parts is subject to high maintenance. While centrifuges are effective at removing suspended solids, they do not account for dissolved solids and heavy metal species in solution. The effluent from a centrifuge would need further treatment prior to disposal. Figure 1.1 shows example of centrifuge (Moulder, 1991).

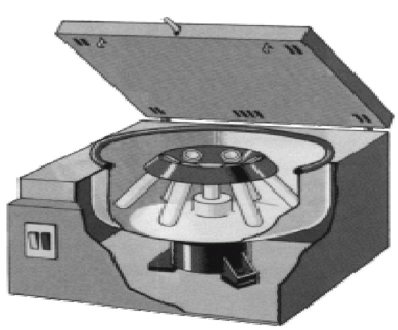

Figure 1.1: Centrifuge (Ahmad, 1994).

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### 1.1.2 Rotary Drum Vacuum Filter

Rotary drum vacuum filter is quite effective at rejecting large solids. Sometimes filtrate must be sent back around to get all of the smaller particles. Usually employs coarse filtration. Vacuum filters require large floor areas and have high capital costs. Figure 1.2 shows example of rotary drum vacuum filter.

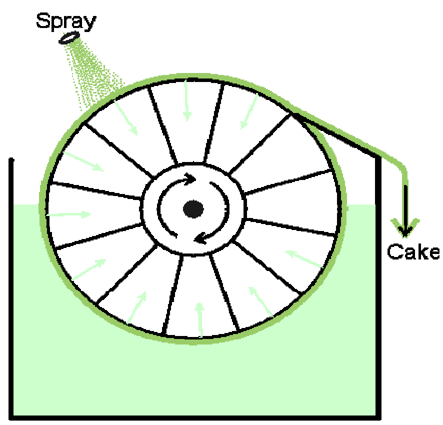


Figure 1.2: Rotary Drum Vacuum Filter (Ahmad, 1994).

### 1.1.3 Dissolved Air Flotation (DAF)

Large tanks where air is bubbled into the bottom and with the use of flocculants, solids are floated to the top and skimmed off. A very large tank is required due to the residence time required. Also chemical addition is a daily if not hourly process and is a significant operating cost. Figure 1.3 shows example of dissolved air flotation system.

Figure 1.3: Dissolved Air Flotation (Ahmad, 1994).

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### 1.1.4 Slope Plate Clarifiers

The process relies on gravity to drop out heavy solids. Here again colloidal materials with small mass and dissolved constituents do not settle. Sometimes it is used in conjunction with flocculation chemicals. These chemicals have limited effect in dropping out heavy metals, BOD, and COD. Figure 1.4 shows example of slope plate clarifier.

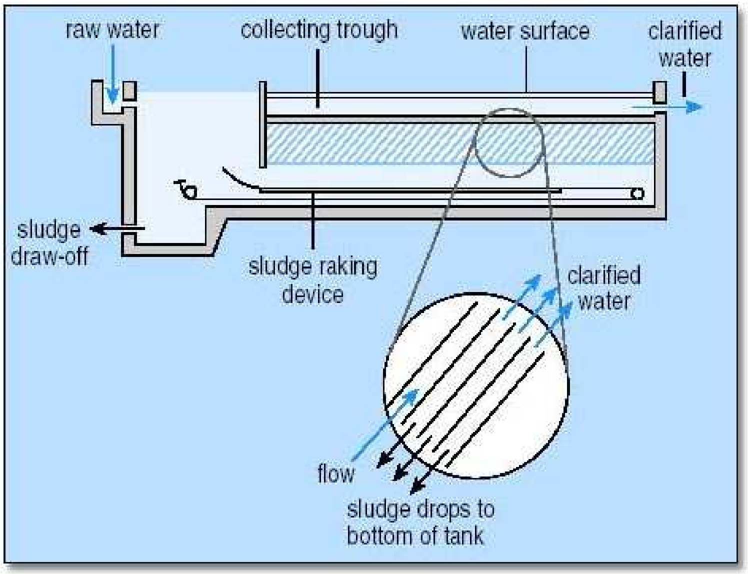


Figure 1.4: Slope Plate Clarifier (Zakaria, 1994).

### 1.1.5 Biological Treatment

This process relies on biological activity to digest the solids in the wastewater. The problem is that the system is extremely temperature and pH sensitive. Also loading must be done at a set rate. The operation of this kind of system usually requires a very skilled operator. It also can take up a lot of floor space due to the amount of residence time required for the bugs to digest the materials (Ahmad, 1997).

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### 1.1.6 Evaporators

It can reduce wastewater to dry solids that can be land filled. Of course water re-use is not possible. Evaporators have very high capital costs and consume huge amounts of energy even for the most efficient models. Figure 1.5 shows example of evaporator.

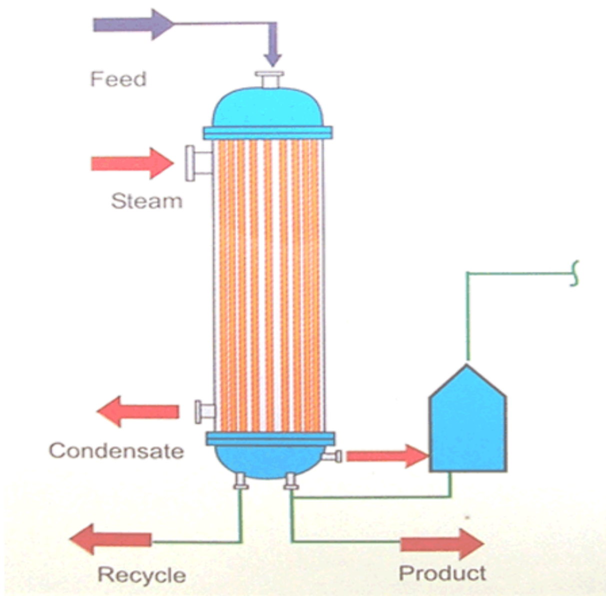


Figure 1.5: Evaporator (Zakaria, 1994).

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## 1.2 Problem Statement

Most communities generate wastewater from both residential and nonresidential sources. Most of the Waste waters containing oily pollutants come from different industrial activities such as mining, power plants, plating facilities and electrical equipment manufacturing. All oily waste water is toxic and non-biodegradable and should be separated from waste waters.

Recently, a number of studies were carried out on low cost treatment from natural resources. The use of low cost treatment for oily wastewater derived from natural resources. Such a low cost treatment is using chitosan membrane which is a biodegradable and biocompatible polymer, produced by deacetylation of chitin. The molecular structure of chitosan is shown in Figure 1.6.

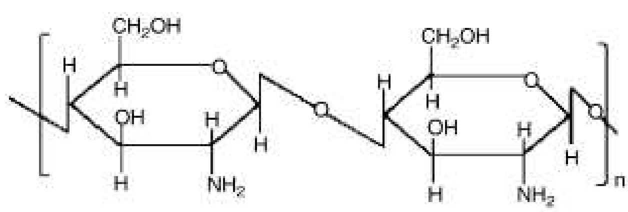


Figure 1.6: Molecular Structure of Chitosan (Younssi, 1994)

Chitin is a structural polysaccharide of crustaceans, insects and some fungi and is the most available biopolymer after cellulose. Chitosan possesses anti-microbial, anti-acid and metalion adsorbing properties which results in its utilization in many industrial applications. One of the applications of chitosan and its derivatives is for separation of oil from waste water. These studies will show that chitosan membrane can be used as a greener method for treating oily wastewater (Ahmad, 1994).

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## 1.3 Objective

The objective for this study is to fabricate chitosan membrane and to investigate the effect of different evaporation times on membrane performance in oily waste water treatment.

## 1.4 Scope of Research

The scopes of this research are:

a) To fabricate chitosan membrane,

b) To Study the performance of chitosan membrane in oily waste water based on the effect of evaporation time while preparing the membrane,

c) To determine the composition of oil left in the water after treatment.

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# CHAPTER 2

# LITERATURE REVIEW

## 2.1 Membranes Separation Processes

Membrane separation technology has been around for many years. Initially, the use of membranes was isolated to a laboratory scale. However, improvements over the past twenty years have made it possible to use membranes on an industrial level. A membrane is simply a synthetic barrier, which prevents the transport of certain components based on various characteristics. Membranes are very diverse in their nature with the one unifying theme to separate. Membranes can be liquid or solid, homogeneous or heterogeneous and can range in thickness. They can be manufactured to be electrically neutral, positive, negative or bipolar. These different characteristics enable membranes to perform many different separations from reverse osmosis to micro-filtration. There are four main categories of membrane filtration. These are determined by the pore size. The following tables give an overview and a classification of membrane separation processes.

Table 2.1 shows size of materials retained, driving force, and type of membrane for various membrane separation processes. Table 2.2 shows examples of applications and separation processes which compete with the respective membrane separation process.

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**Table 2.1: Size of materials retained, driving force, and type of membrane (Mohr, 1989).**

| Process | Size of materials retained | Driving force | Type of membrane |
|---|---|---|---|
| Microfiltration | 0.1 - 10 µm microparticles | Pressure difference (0.5 - 2 bar) | Porous |
| Ultrafiltration | 1 - 100 nm macromolecules | Pressure difference (1 - 10 bar) | Microporous |
| Nanofiltration | 0.5 - 5 nm molecules | Pressure difference (10 - 70 bar) | Microporous |
| Reverse Osmosis | < 1 nm molecules | Pressure difference (10 - 100 bar) | Nonporous |

**Table 2.2: Examples of applications and alternative separation processes (Mohr, 1989).**

| Process | Applications | Alternative Processes |
|---|---|---|
| Microfiltration | Separation of bacteria and cells from solutions | Sedimentation, Centrifugation |
| Ultrafiltration | Separation of proteins and virus, concentration of oil-in-water emulsions | Centrifugation |
| Nanofiltration | Separation of dye and sugar, water softening | Distillation, Evaporation |
| Reverse Osmosis | Desalination of sea and brackish water, process water purification | Distillation, Evaporation, Dialysis |

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